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SUMMARY

Creep tests of 1000 hours duration and greater were conducted in the temperature range 2000° to 2200° F on a commercially available columbium alloy (FS-85) in vacuum units designed and built to achieve ultimate pressures of 10^{-9} torr. At a stress level of 10 000 pounds per square inch, FS-85 exhibited moderate creep resistance at 2000° F, 1-percent strain being achieved in 775 hours. At 2100° and 2200° F and the same stress level, the alloy was much weaker, 1-percent strain being achieved in 355 and 50 hours, respectively. The Manson-Haferd linear time-temperature parameter proved moderately successful in predicting the long-time creep lives from relatively short-time creep data, the maximum error between predicted and experimental lives being a factor of 1.7 and the average error being a factor of 1.3. The agreement is considered good in view of the large scatter generally observed in long-time high-temperature creep tests. In all cases, however, the observed experimental life was shorter than the predicted life. The nonconservative nature of these predictions may be due in part to the fact that the predictions were based on data obtained at a pressure level of approximately 10^{-6} to 10^{-7} torr, but the long-time tests to which the predictions are compared were made at a lower pressure level, 10^{-8} to 10^{-9} torr.

Creep tests conducted in the conventional-vacuum units (10^{-6} torr) and the ultrahigh-vacuum units under the same conditions of temperature and stress yielded creep curves that are virtually identical for the first few hundred hours but that deviate with increasing test time. The conventional-vacuum tests then showed a reduced creep rate compared with that of the ultrahigh-vacuum tests.

INTRODUCTION

As previously reported (ref. 1), columbium alloys are of interest as containment materials for alkali liquid metals in advanced space electric-power systems. Because

this application involves both long operating times (10 000 hr or more) and high temperatures (1800° to 2400° F), creep strength of the containment materials is of prime concern. Unfortunately, very little information on the creep properties of potentially useful materials is currently available. As part of a larger program aimed at identifying the most promising materials for this application and characterizing their properties, the creep behavior of a highly fabricable, commercially available columbium alloy, FS-85 (Cb + 28Ta + 10W + 1Zr), was investigated over the temperature range 1800° to 2800° F in vacuum (10^{-5} to 10^{-9} torr) for times up to 2500 hours.

The test program was devised to utilize the Manson-Haferd linear time-temperature parameter (ref. 2) to determine its applicability for predicting long-time creep behavior from relatively short-time tests and to aid in characterizing the long-time creep properties of FS-85 with a minimum expenditure of testing time and an efficient use of test facilities.

MATERIAL

The material used in this study was commercially available 0.030- by 12- by 24-inch FS-85 sheet procured in the as-rolled condition. The manufacturer reported that the material had been cold-rolled 50 percent following the last in-process recrystallization heat treatment. The chemical analysis of the as-received sheet is shown in table I.

TABLE I. - CHEMICAL ANALYSIS OF AS-RECEIVED SHEET

Element	Tanta- lum	Tung- sten	Zirco- nium	Oxygen	Nitro- gen	Carbon	Hydro- gen	Colum- bium
Composition, percent by weight	27.76	9.49	0.80	0.0180	0.0042	0.0060	0.0002	Balance

Creep specimens having a 1-inch gage length and a 1/4-inch gage width (fig. 1) were machined from the as-received sheet with the specimen axis parallel to the final rolling direction. The specimens were evaluated in the recrystallized condition. Figure 2 shows the microstructure resulting from the recrystallization heat treatment, 1 hour at 2600° F, in a vacuum of 10^{-6} torr. The recrystallized material had an average grain diameter of 0.019 millimeter and a Vickers hardness of 189 (10-kg load) for the major surface. Specimens were weighed to the nearest 0.1 milligram before and after the recrystallization heat treatment to note any possible contamination resulting from heat treatment before creep testing. No significant weight changes were observed; that is, the maximum contamination observed amounted to a maximum of 15 parts per million.
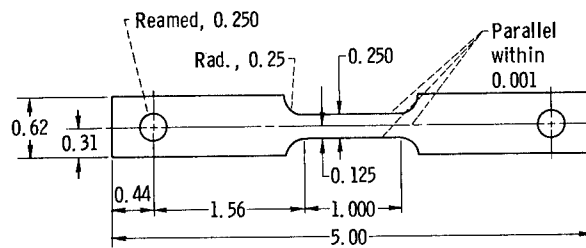
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Figure 1. - Standard sheet creep-test specimen. (All dimensions are in inches.)
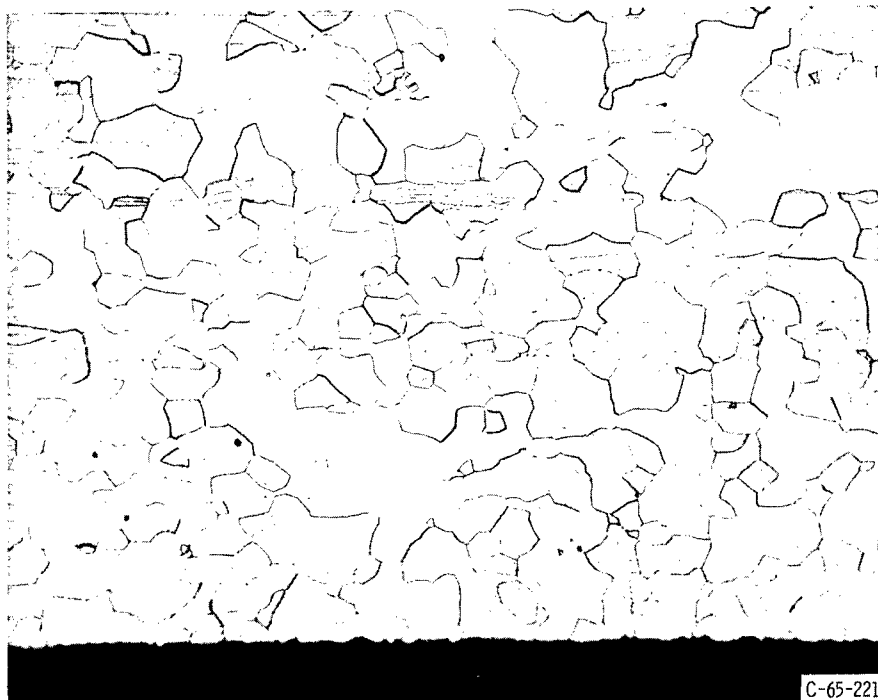

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Figure 2. - Microstructure of recrystallized FS-85 sheet before creep testing. Etchant, equal parts of nitric and hydrofluoric acids modified with sulfuric acid, hydrogen peroxide, and acetic acid. X250.

APPARATUS

Two types of creep units differing in their ultimate vacuum capability were used in this program. For the initial phase of the program, in which tests were generally limited to a maximum of about 400 hours, relatively conventional vacuum creep units with an ultimate vacuum capability of about 5×10^{-7} torr were used. For the second phase of the program, which involved tests up to about 2500 hours, new creep units capable of achieving an ultimate vacuum of 10^{-9} torr were designed and built. [Both types of units are briefly described including test procedures.]

Conventional High-Vacuum Creep Unit

The major features of the creep equipment used for the short-time tests (up to a few
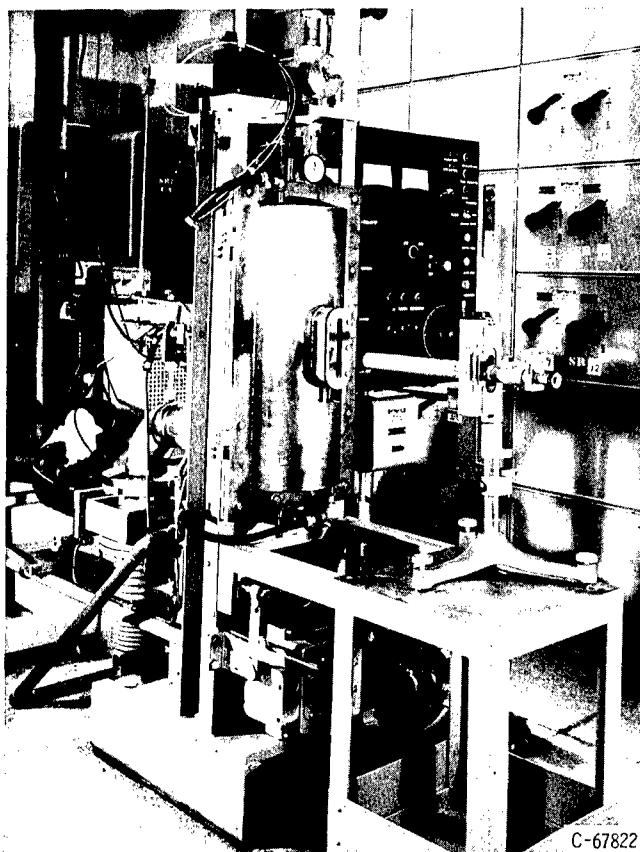
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Figure 3. – Conventional vacuum creep-rupture unit with cathetometer clamped in viewing position.

hundred hours) are described in reference 3. This equipment, however, was upgraded extensively, as described in the following paragraph, to achieve vacuums in the 10^{-7} torr regime to minimize atmospheric contamination in creep testing of columbium alloys. Vacuum requirements for creep testing of columbium alloys at elevated temperatures are discussed in reference 1. Pertinent information on contamination of refractory metals by residual gases in oil-diffusion-pump vacuum systems is reported in reference 4.

The test chamber (fig. 3) is a water-cooled stainless-steel chamber that is split longitudinally and hinged on one side. All the elastomeric seals are made of Viton to minimize outgassing. The sliding seal previously used to permit external lever loading was eliminated because dead-weight loading with stainless-steel weights contained within the vacuum chamber was used. Gate valves were eliminated from the pumping systems as likely outgassing and leak sources.

The chamber is evacuated by a two-stage mechanical forepump rated at 15 cubic feet per minute (8 liters/sec) and backing a three-stage fractioning oil-diffusion pump having a maximum pumping speed of about 300 liters per second when baffled. Chevron baffles placed between the diffusion pump and the chamber are cooled continuously and automatically by liquid nitrogen to minimize back diffusion of pump-oil vapors. Liquid nitrogen is supplied to the baffles by a vacuum-jacketed pipeline from a self-pressurizing 2500-gallon Dewar. The test chamber can be evacuated to 10^{-7} torr, but outgassing of the specimen and chamber during initial heating causes the pressure to rise. Consequently, in most tests, the pressure at the beginning of the test was in the mid 10^{-6} torr range and gradually decreased to below the mid 10^{-7} torr range as the test proceeded. Pressure was measured by means of a hot-cathode ionization gage located at the top of the test chamber.

Details of the heater assembly are shown in figure 4. The heater consists of a 0.020-inch-wall seamless tantalum tube $1\frac{1}{2}$ inches in diameter and 7 inches long that is

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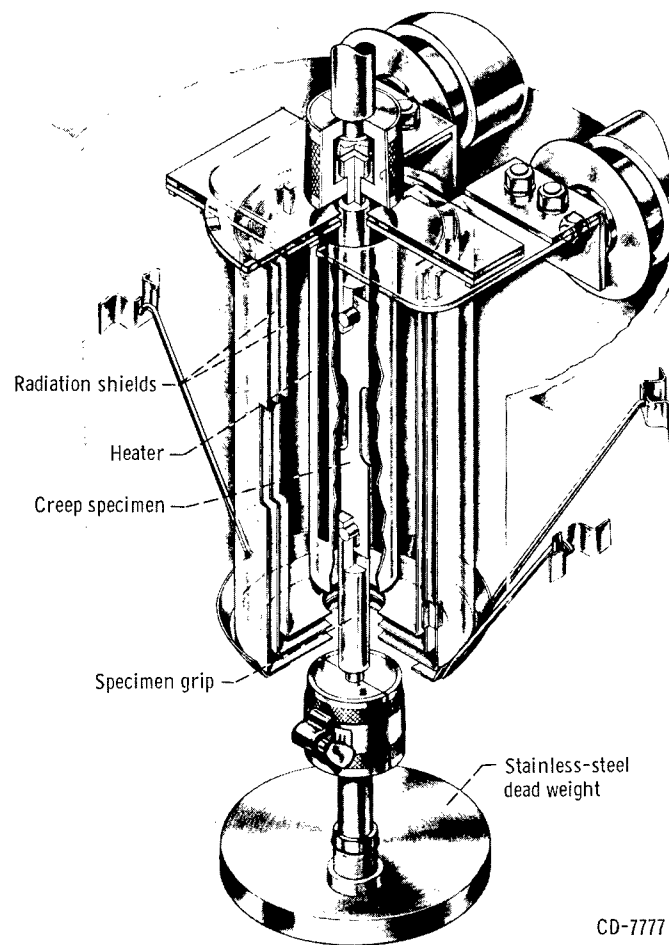
Figure 4. – Cutaway drawing of heater assembly and pin-type gripping method used in conventional vacuum unit.

slotted longitudinally to within 1 inch of the bottom and welded at the top to 0. 100-inch tantalum connectors. Tantalum radiation shields completely surround the heater. Pin-type grips were employed in these tests, the sheet specimen being pinned to the grips within the heating element as shown in figure 4. The temperature gradient along the $1\frac{1}{2}$-inch reduced section of the specimen was less than 6° F over the 1800° to 3000° F temperature range used for the tests reported herein. The axial loading fixture and the split connectors are identical to those described in reference 3. Temperature of the specimen was measured with either platinum-13-percent-rhodium/platinum or tungsten/tungsten-26-percent-rhenium thermocouples tied to the specimen surface at the gage center. The former was used for temperatures from 1800° to 2400° F, and the latter was used at higher temperatures. The temperature was held constant within 0. 5 percent of the desired temperature by using a 15-kilowatt electronic voltage regulator capable of controlling the voltage to 0. 1 percent of the desired voltage.
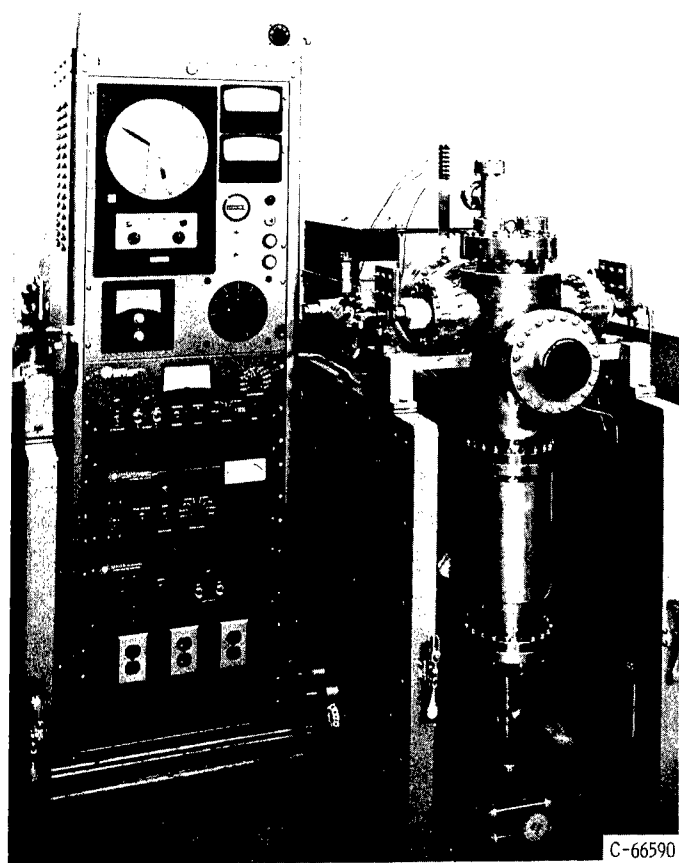
Figure 5. - Ultrahigh-vacuum creep unit.

Ultrahigh-Vacuum Creep Unit

Creep equipment (figs. 5 and 6) capable of achieving pressures of 10^{-8} torr and lower was designed for long-time tests (1000 hr and greater). The major features of this equipment are reported in reference 1. It consists of a bakable stainless-steel chamber utilizing oxygen-free, high-conductivity (OFHC) copper gaskets for all demountable seals, which is evacuated by a 400-liter-per-second sputter ion pump. Dead-weight loading with tungsten weights contained within the vacuum chamber is utilized. Power is supplied to the tubular tantalum heater from a 15-kilowatt saturable core reactor. The temperature is measured with platinum-13-percent-rhodium/platinum thermocouples and is controlled by a proportioning-type controller capable of regulating the desired temperature to within $\pm 4^\circ$ F. Pressure is measured by means of a nude hot-cathode ionization gage, which is located in the 6-inch-diameter pumping line on the back of the test chamber.

PROCEDURE

Before creep testing, fiducial marks, in the form of Knoop hardness impressions, were placed near the ends of the reduced section of the creep specimen, 1.000 inch apart. High-vacuum technology requires that the specimen be thoroughly washed in acetone and handled only with lint-free nylon gloves from this point on.

The appropriate thermocouple was tied to the center of the gage section with tantalum wire. The entire reduced section of the specimen was wrapped with 2-mil tantalum foil into which two sight holes had been cut so that the fiducial marks were visible. This tantalum foil shield was used to help prevent contamination of the specimen from possible backstreaming of diffusion-pump oil vapors in the conventional units or furnace chamber outgassing in the ultrahigh-vacuum units (ref. 4) and to provide a reliable temperature
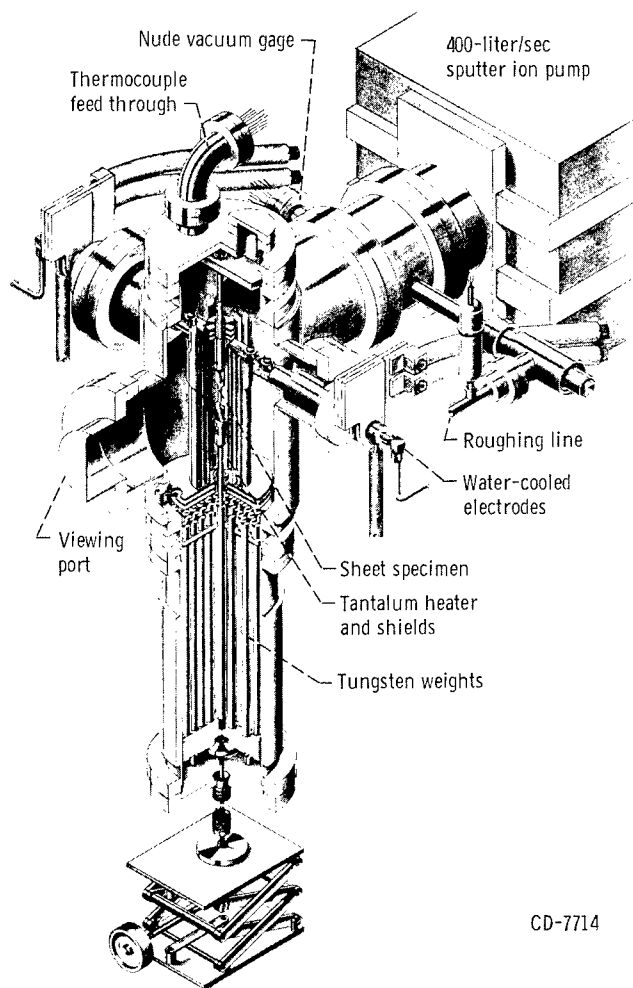
Figure 6. - Cutaway drawing of ultrahigh-vacuum creep unit.

reading of the specimen by shielding the thermocouple from direct radiation from the heater.

Following the thermocouple assembly, the specimen was loaded into the respective test chamber, and the deadweight load was attached and supported. Internal loading utilized in this manner allows for high accuracy so that the actual stress is always within ± 1 percent of the desired stress, and the probability of nonaxial loading is minimized. During evacuation, leak checking with a helium mass spectrometer, bakeout of the ultrahigh-vacuum chamber, and heating of the specimen to the test temperature, the weights were supported on a pedestal that could be retracted through a double 0-ring seal on the conventional creep chamber or by a metallic-bellows seal on the ultrahigh-vacuum creep chamber. In the conventional creep unit the specimen was usually brought to temperature in 1 hour with a corresponding pressure rise of about 5×10^{-6} torr.

The pressure generally fell below 5×10^{-7} torr a few hours after loading. The ultrahigh-vacuum unit required approximately 30 hours to reach a temperature of 2000° F. The pressure was limited to about 5×10^{-7} torr during heatup by controlling the heating rate. After 10 to 15 hours at temperature, the pressure was generally below 5×10^{-8} torr, and it continued to decrease slowly as the test proceeded. In all instances, a micro-optical pyrometer was used to measure the surface-brightness temperature at the fiducial marks before loading and at the end of the test. Such measurements were used to check the temperature stability indicated by the thermocouples.

Creep Strain Measurements

Creep strain was measured optically with a cathetometer clamped to the vacuum

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chamber frame as shown in figure 3. After the specimen had been stabilized at the desired temperature, an initial gage length was read prior to loading. The strain on loading was read and is incorporated in the reported total creep strain readings. Precision of the total creep strain measured optically with the cathetometer is estimated to be ±0.04 percent.

Selection of Temperature and Stress Levels

As previously indicated, the creep test program was conducted in two phases. For the initial phase, creep tests were limited to a maximum time of about 400 hours. This limitation was imposed by the likelihood of contamination of the specimen in long-time tests by residual gases in the available conventional-vacuum creep units. (The extent of contamination actually experienced in these tests will be described in the section on p. 12.) For the second phase of the program, equipment with greatly improved vacuum capability was available, and creep testing up to several thousand hours was possible. Thus the primary purpose of the first phase of this study was to obtain creep data from relatively short-time tests that would guide selection of stress levels for the longer tests of the second phase. The type of information ultimately desired is allowable design stress data for total strain values of between 1 and 5 percent in 10 000 hours over the temperature range 1800° to 2400° F. Tests of 10 000 hours were considered beyond the scope of this investigation because FS-85 is only one of several columbium alloys under consideration for use in advanced-electric-space power systems.

In view of the need to predict long-time creep behavior from the results of relatively short-time tests, such as those in the first phase of this program, the time-temperature parameter method of extrapolating creep data was utilized. For this purpose, the Manson-Haferd linear parameter (ref. 2) was adopted and a test program to determine the parametric constants and the master curve for the parameter was set up following the guidelines suggested in reference 5. Stress levels of 25 000 and 4000 pounds per square inch were selected as limiting stresses so that the maximum difference in slopes was provided when the log of the test time t in hours was plotted as a function of test temperature T in $^{\circ}$F. The intersection of these constant-stress lines determines the parametric constants T_a and t_a of the linear parameter

$$P = \frac{T - T_a}{\log t - \log t_a}$$

The master curve, stress against the parameter P, is established from the slopes of the semilog plots of time for an allowable creep strain at constant stress against test temperature from the first phase of this program.

In the second phase of the program, creep behavior under five previously undetermined stress-temperature conditions was predicted by using the linear parameter, and creep tests were conducted in the ultrahigh-vacuum creep units to check the reliability of the predictions.

RESULTS AND DISCUSSION

Creep Tests in Conventional Creep Units

The results of the creep tests performed in the conventional vacuum units at pressures of 10^{-6} to 10^{-7} torr are shown in figure 7. The total creep curve is not presented; generally tests were terminated either after the specimen had exceeded 5-percent creep strain or after 300 to 400 hours if creep rates were too low to result in 5-percent strain in that time period. A few tests were permitted to run to rupture. The resulting rupture times and elongations at fracture are shown in table II. Because FS-85 is a very ductile

TABLE II. - RUPTURE TIMES AND ELONGATIONS

AT FRACTURE FOR FS-85 TESTED AT VARIOUS

CONDITIONS OF TEMPERATURE AND STRESS

Specimen	Test conditions		Rupture time, hr	Elongation in 1 inch, percent
	Temperature, $^{\circ}$F	Stress, psi		
FS-85-8	2620	4 000	214.0	92.0
FS-85-9	2800	4 000	43.7	53.0
FS-85-1B	2400	10 000	39.7	70.0
FS-85-10B	1900	25 000	50.5	42.0
FS-85-7B	2000	25 000	6.5	41.0

alloy, the allowable creep strain rather than the rupture life will be the important design criterion for most high-temperature structural applications of this alloy.
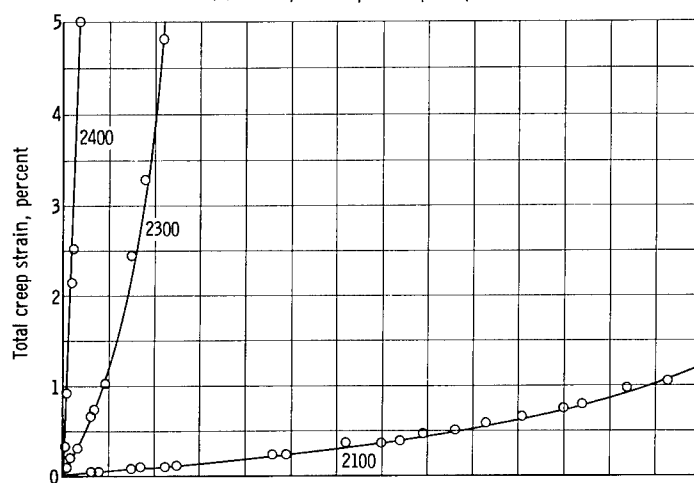
Determination of Manson-Haferd Linear
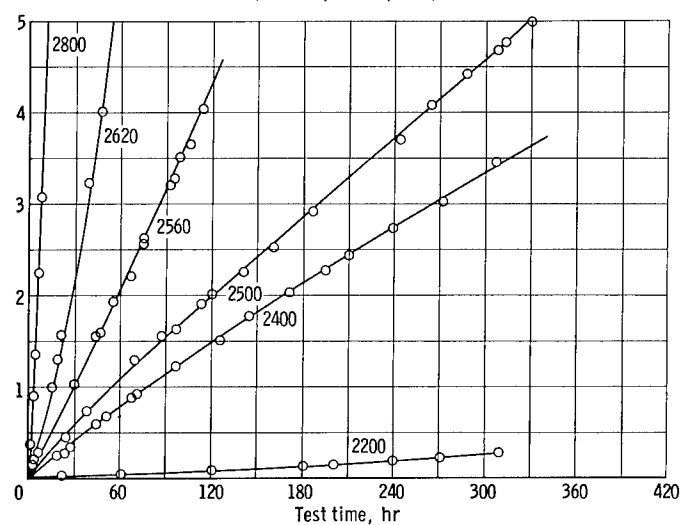
Time-Temperature Parameter

Creep tests covering a wide range of temperature, 1800° to 2800° F, were conducted at three constant stress levels, 25 000, 10 000, and 4000 pounds per square inch, to
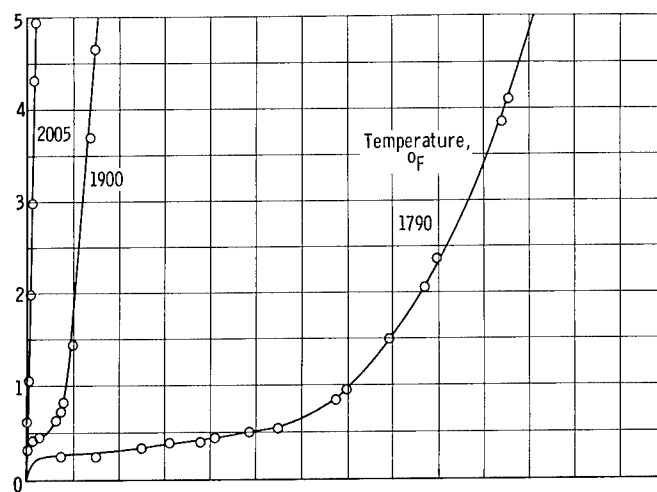
(a) Stress, 25 000 pounds per square inch.

(b) Stress, 10 000 pounds per square inch.

(c) Stress, 4000 pounds per square inch.

Figure 7. – Creep behavior of FS-85 at high temperatures. Pressure, approximately 10^{-7} torr.

(a) Total creep strain, 1 percent.

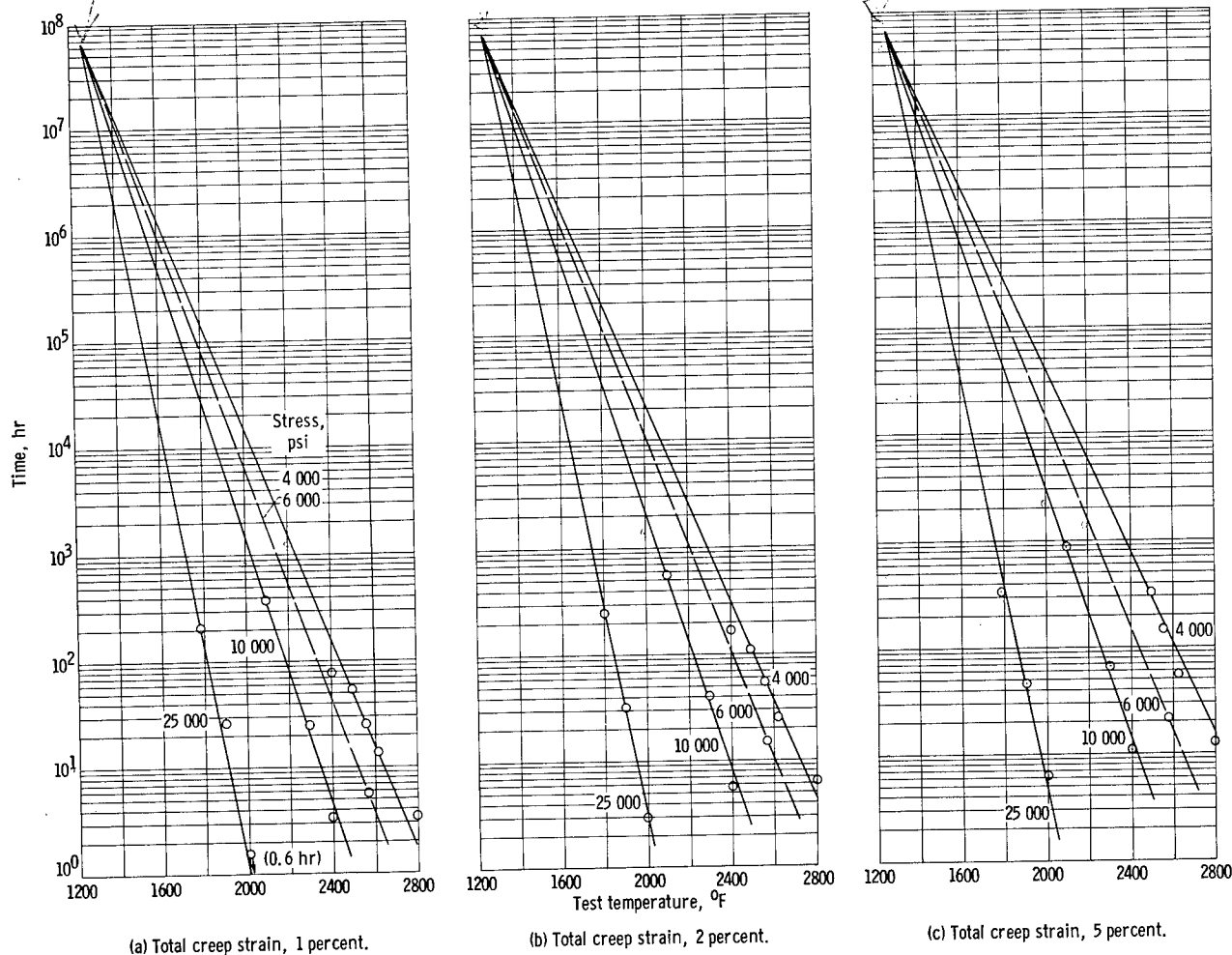(b) Total creep strain, 2 percent.

(c) Total creep strain, 5 percent.

Figure 8. - Time-temperature constant stress curves for FS-85.

facilitate the determination of the linear time-temperature parameter. From the creep curves shown in figure 7, the times corresponding to total strains of 1, 2, and 5 percent were replotted as functions of temperature in figure 8. To determine the parametric constants T_a and t_a, straight lines were drawn through each set of constant stress values (i. e., 25 000, 10 000, and 4000 psi) to a point of common intersection. The coordinates of this point, T_a and t_a, are, respectively, 1260° F and 6.5×10^7 hours (log $t_a = 7.813$). Although the coordinates of this point were determined visually, the uncertainties in determining the parametric constants associated with this procedure generally do not significantly affect the resulting predictions. As has been pointed out (ref. 6), the accuracy of a correlation or an extrapolation depends on the combination of the parametric constants not on the individual values. Nearly the same degree of accuracy can be obtained with several sets of constants even though numerically the parametric constants may differ appreciably. Following the determination of the parametric constants, an additional test at 6000 pounds per square inch was conducted to better
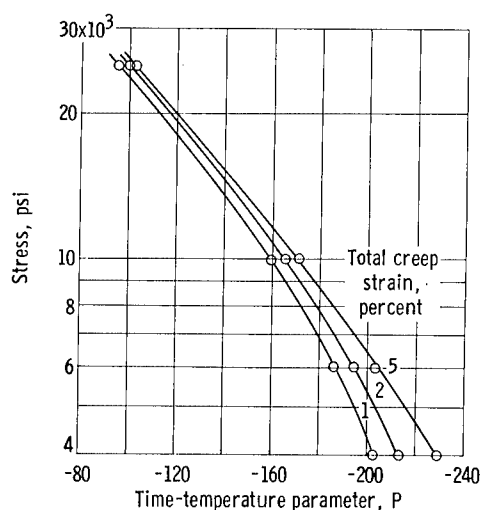
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Figure 9. – Master curve of linear time-temperature parameter.

define the master curves at low stress levels. The master curves for 1-, 2-, and 5-percent total creep strains, constructed from the slopes of the constant-stress lines of the semilog plots of time against temperature and the preceding constants T_a and log t_a, are shown in figure 9. Theoretically it is now possible to calculate and predict the times required to produce 1-, 2-, and 5-percent creep strains if the nominal stresses used are in the range 4000 to 25 000 pounds per square inch and the test temperatures are in a range where major metallurgical transformations are not operative. If the linear parameter is applicable to predicting long-time creep strain, preliminary design data presently sought, such as the allowable stress to limit total creep strain to 1 percent in 10 000 hours at high temperatures, would be available. Figure 10 shows design curves based on the linear time-temperature parameter, which indicate predicted times for 1-, 2-, and 5-percent strains as functions of stress at 2000° and 2200° F, respectively. Based on these plots, the allowable stress for a 1-percent strain of FS-85 in 10 000 hours at 2000° F would be 5000 pounds per square inch. At 2200° F, it is presently not possible to predict this allowable stress because it is well below the lower limit of the master curves (4000 psi), and thus the prediction would not be valid. The allowable 10 000-hour stress level necessary to limit total creep strain to 1, 2, and 5 percent is shown in figure 11, a plot also based on the linear time-temperature parameter.

Extent of Oxygen Contamination During Creep Testing in Conventional Units

The foregoing analysis of the creep data must be viewed with reservation because contamination of the test specimens with oxygen occurred during testing in the conventional creep units despite the stringent precautions taken to achieve low ultimate pressures and low leak rates. The extent of contamination, measured by vacuum-fusion analysis of a portion of the specimen gage section after testing, is shown in table III. In general, the increase in oxygen content during tests of less than about 400 hours was limited to a maximum of about 200 parts per million. In a few tests, contamination was significantly greater than the preceding value, but was usually associated with a power failure or a vacuum leak that caused the test to be terminated.

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(a) Temperature, 2000° F.
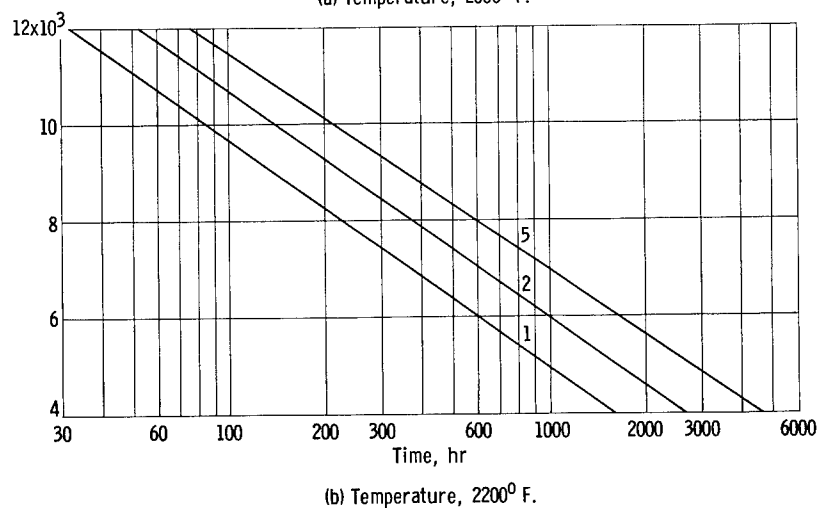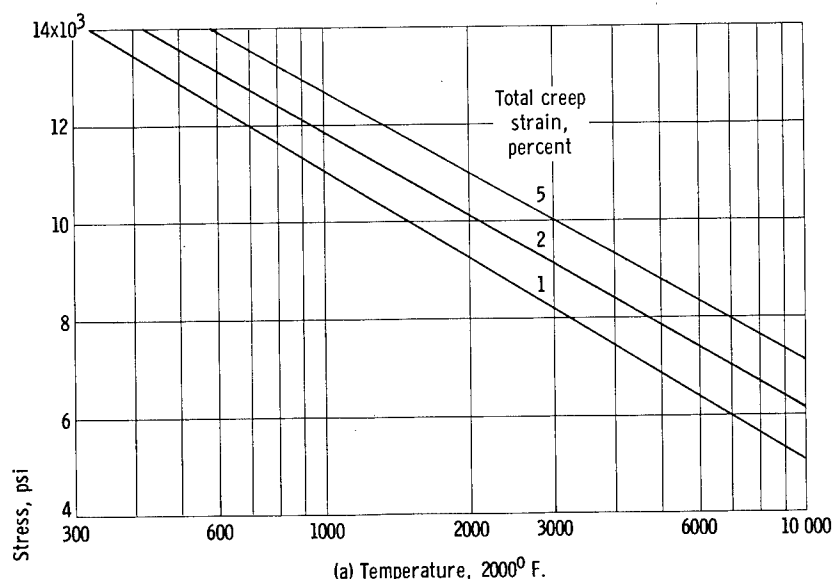

(b) Temperature, 2200° F.

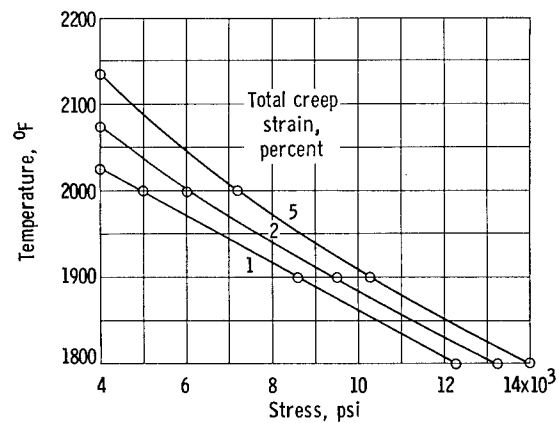Figure 10. - Design curves based on linear time-temperature parameter.

Figure 11. - Predicted 10 000-hour stress levels as function of temperature for various creep strains.

TABLE III. - RESULTS OF VACUUM FUSION ANALYSIS ON CONVENTIONAL

VACUUM CREEP SPECIMENS AFTER TESTING

Specimen	Test conditions					Increase in oxygen content during creep testing, ppm
	Time, hr	Temperature, °F	Stress, psi	Pressure, torr		
				Start of test	End of test	
	(a)					(b)
FS-85-1	309	2200	4 000	9×10^{-6}	1.4×10^{-6}	[c]306
FS-85-6	309	2400	4 000	9×10^{-6}	4×10^{-7}	108
FS-85-8B	425	2500	4 000	2×10^{-5}	5×10^{-7}	160
FS-85-4C1	115	2560	4 000	6×10^{-6}	6×10^{-7}	[d]1020
FS-85-8	214	2620	4 000	9×10^{-6}	3×10^{-7}	90
FS-85-9	44	2800	4 000	3×10^{-5}	4×10^{-6}	120
FS-85-13	24	2575	6 000	6×10^{-6}	1×10^{-6}	90
FS-85-2C1	926	2100	10 000	3×10^{-5}	3×10^{-7}	[e]195
FS-85-1C1	92	2300	10 000	8×10^{-6}	4.5×10^{-7}	80
FS-85-1B	40	2400	10 000	2×10^{-5}	7×10^{-7}	120
FS-85-4B	405	1790	25 000	5×10^{-6}	3×10^{-7}	180
FS-85-10B	66	1900	25 000	2×10^{-5}	6×10^{-7}	[f]380
FS-85-7B	12	2000	25 000	2×10^{-5}	2×10^{-6}	60

[a]Time at temperature, or total duration of test run.

[b]Baseline level in as-received sheet, 180 ppm oxygen.

[c]No liquid nitrogen available for last 100 hr of test; pressure rose from 4.0×10^{-7} to 1.4×10^{-6} torr when liquid-nitrogen supply was exhausted.

[d]Test terminated because of complete power failure; contamination probably resulted after creep data were gathered.

[e]Power failure early in test; testing resumed immediately.

[f]Test run to rupture; vacuum fell to 0.5 mm immediately after rupture.

The effects of oxygen contamination at the indicated levels on the high-temperature creep rates of FS-85 are unknown and must await further study. In a study of the effects of the degree of vacuum on the creep behavior of a columbium - 0.6-percent-zirconium alloy at 1000° C (1832° F) (ref. 7), changes in alloy creep behavior were correlated with residual-gas contamination in the vacuum system. In these tests, however, the oxygen contamination levels were very much larger than those observed in the tests reported herein.
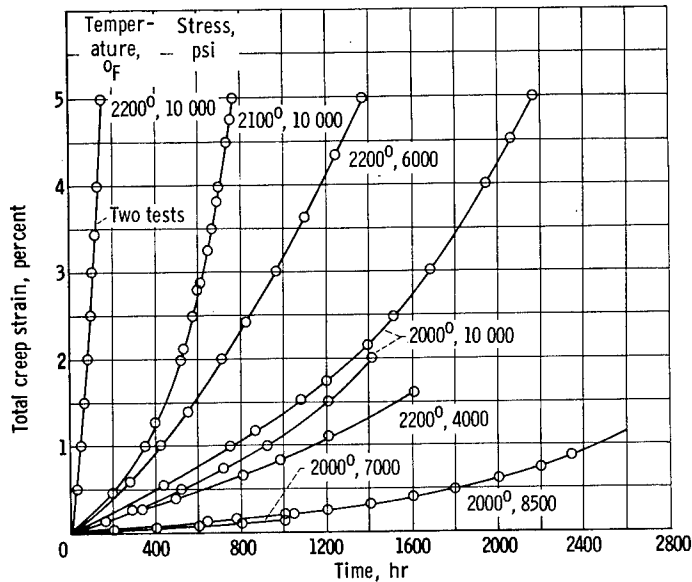
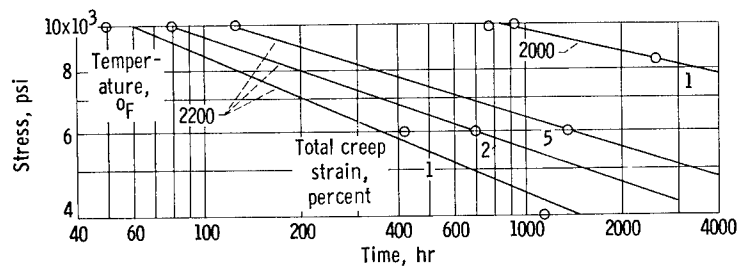Figure 12. - Ultrahigh-vacuum creep behavior of FS-85.

Figure 13. - Effect of stress on time to achieve desired strain.

Creep Tests in Ultrahigh-Vacuum Creep Units

Relatively few creep tests of FS-85 have been conducted in the ultrahigh-vacuum units; the available data are shown in figure 12. The two tests conducted at 2000° F and 10 000 pounds per square inch were duplicate tests and afford a measure for scatter of the creep data in these tests.

The creep curves of figure 12 indicate that FS-85 has moderate creep strength at 2000° F but that its strength decreases rapidly with increasing temperature. For example, at a stress level of 10 000 pounds per square inch, 1-percent creep strain was achieved in 775 hours at 2000° F, 355 hours at 2100° F, and 50 hours at 2200° F. The stress dependence of the time to achieve 1-, 2-, and 5-percent total strain at 2000° F and 1-percent strain at 2200° F is more readily compared in figure 13. For example, the curve for 1-percent total strain at 2200° F indicates that decreasing the stress by a factor of 2, from 10 000 to 5000 pounds per square inch, increases the time for 1-percent

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TABLE IV. - COMPARISON OF TIME PREDICTED BY LINEAR TIME-TEMPERATURE

PARAMETER WITH EXPERIMENTAL TIME TO ACHIEVE TOTAL CREEP

STRAINS OF 1, 2, AND 5 PERCENT

Test conditions		1-Percent creep strain		2-Percent creep strain		5-Percent creep strain	
Temperature, ^{o}F	Stress, psi	Time, hr					
		Experi- mental	Predic- ted	Experi- mental	Predic- ted	Experi- mental	Predic- ted
2000	10 000	[a]835	1422	[b]1372	2255	2175	3045
2000	8 500	[c]2480	3042	-----	----	----	----
2100	10 000	355	375	500	567	765	800
2200	4 000	[c]1140	1360	-----	----	----	----
2200	6 000	425	567	710	985	1370	1710

[a]Average of two tests; 775 and 900 hr.

[b]Average of two tests; 1325 and 1420 hr.

[c]Test terminated after 1-percent creep strain.

strain by a factor of 13.8, from 50 to about 690 hours. Based on the limited data available, the stress dependency of the time to achieve 1-percent strain is somewhat larger at 2000° F than at 2200° F.

Comparison of Ultrahigh-Vacuum Creep Data With Predictions

Based on Linear Time-Temperature Parameter

As mentioned in the INTRODUCTION, the selection of the temperature and stress levels used for the long-time tests in the ultrahigh-vacuum creep units was made with the aid of the linear time-temperature parameter determined from the short-time tests conducted in the first phase of this study. Thus, the long-time tests afforded a means of experimentally evaluating the reliability of this parameter as used for predicting long-time creep behavior. The predicted and experimental times for 1-, 2-, and 5-percent total strain under five combinations of temperature and stress are compared in table IV. In general, the experimental data are in moderate agreement with the predicted times; that is, the deviation of the predicted times from the experimentally observed times ranged from a factor of 1.05 to 1.7. In view of the fact that creep data frequently show considerable experimental scatter, this agreement is probably as good as could reasonably be expected.

It is disappointing to note that the predicted times are not conservative; that is, the

TABLE V. - RESULTS OF VACUUM FUSION ANALYSIS FOR OXYGEN ON

ULTRAHIGH-VACUUM CREEP SPECIMENS AFTER TESTING

| Specimen | Test conditions | | | | | Increase in oxygen content during creep testing, ppm |
| | Time, hr (a) | Temperature, °F | Stress, psi | Pressure, torr | | |
				Start of test	End of test	(b)
FS-85-14	2212	2000	10 000	7×10^{-7}	6×10^{-9}	60
FS-85-6B	1522	2000	10 000	2×10^{-7}	4×10^{-9}	62
FS-85-23	2566	2000	8 500	2.2×10^{-7}	3.1×10^{-9}	37
FS-85-25	816	2100	10 000	3×10^{-7}	5.1×10^{-9}	58
FS-85-24	210	2200	10 000	5×10^{-7}	1.7×10^{-8}	59
FS-85-21	1393	2200	6 000	7×10^{-7}	9.0×10^{-9}	64
FS-85-22	1670	2200	4 000	7×10^{-7}	1.8×10^{-8}	77

[a]Time at temperature, or total duration of test run.
[b]Baseline level in as-received sheet, 180 ppm oxygen.

experimentally determined life for a given total strain is less than the predicted life in all cases. This suggests that some factor may have been operative in the short-time tests in the conventional creep units that reduced creep rates below those that would have been observed in the ultrahigh-vacuum units under identical stress-temperature conditions. The nonconservative nature of these predictions may be due in part to the fact that the predictions were based on data obtained at a pressure level of approximately 10^{-6} to 10^{-7} torr, but the long-time tests to which the predictions are compared were made at a lower pressure level, 10^{-8} to 10^{-9} torr. Possibly, reduction of creep rates by interstitial contamination in the conventional units and the absence of such an effect in the ultrahigh-vacuum creep units could lead to the observed behavior. Comparison of tables III and V, which list oxygen pickup during testing in the conventional and ultrahigh-vacuum creep units, respectively, indicates that contamination occurred at a greatly reduced rate in the latter unit.

Creep rates for two sets of tests that were conducted in both the conventional and ultrahigh-vacuum units under identical stress and temperature conditions (4000 psi at 2200° F and 10 000 psi at 2100° F) are virtually identical for the first 200 to 300 hours of testing, as shown in figure 14. The 4000 psi, 2000° F tests show only the difference due to strain on loading for the duration of the tests, but the tests at 10 000 pounds per square inch and 2100° F show increasing deviation with increasing time. For example, at 1-, 2-, and 5-percent strains for the tests run at 10 000 pounds per square inch and 2100° F, the deviation of the conventional vacuum test from the ultrahigh-vacuum test was 40, 80, and 100 hours, respectively. Unfortunately, a direct comparison of oxygen content of the
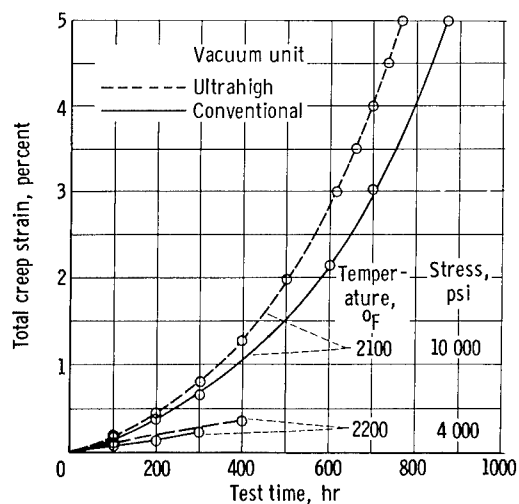
Figure 14. - Comparison of creep curves from tests in conventional and ultrahigh-vacuum creep units.
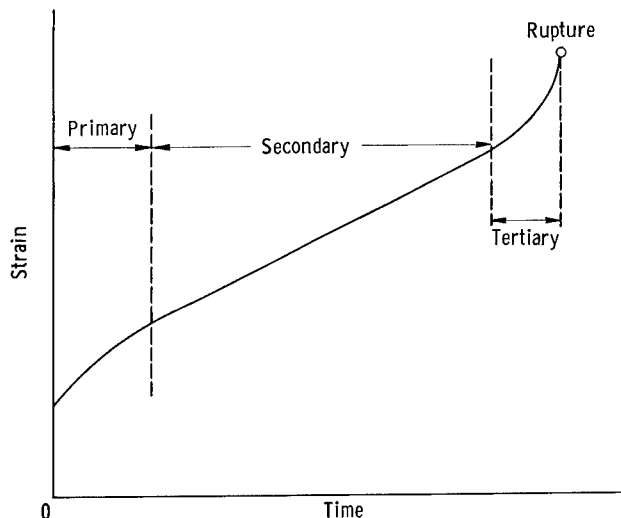

Figure 15. - Typical creep curve.

specimens from these 2100° F tests cannot be made because of a power failure that may have affected the contamination level of the specimen run in the conventional vacuum unit.

## Shape of Observed Creep Curves

Examination of the creep curves obtained for the FS-85 alloy in this study (fig. 7 for conventional units and fig. 12 for the ultrahigh-vacuum unit) indicates that most of these curves exhibit relatively unusual shapes. For comparison, the shape of a typical creep curve, illustrating the three stages of creep generally observed, is shown in figure 15. At the lower stresses used in this study, 4000 and 10 000 pounds per square inch, no primary stage of creep was observed. Because this stage, wherein creep rate decreases continuously with increasing time, is usually attributed to the rate of work hardening exceeding the rate of recovery, its absence suggests that work hardening is very slight and/ or that recovery is very rapid at the low creep rates and relatively high test temperatures used. Even more surprising than the absence of a primary creep stage is the general absence of a secondary or linear creep stage in many of the tests, particularly those conducted in the ultrahigh-vacuum units (fig. 12). In many tests, creep rates appeared to increase continuously with increasing time from the start of the test. This behavior, which commonly characterizes the tertiary creep stage, is usually observed prior to rupture and is frequently associated either with dimensional instability (necking) or structural instability, such as internal void formation.

Although such creep curves are unusual, similar curves have been reported previously for other columbium alloys (ref. 8) at high temperatures and low stress levels.

18

(a) Before test.

C-65-222
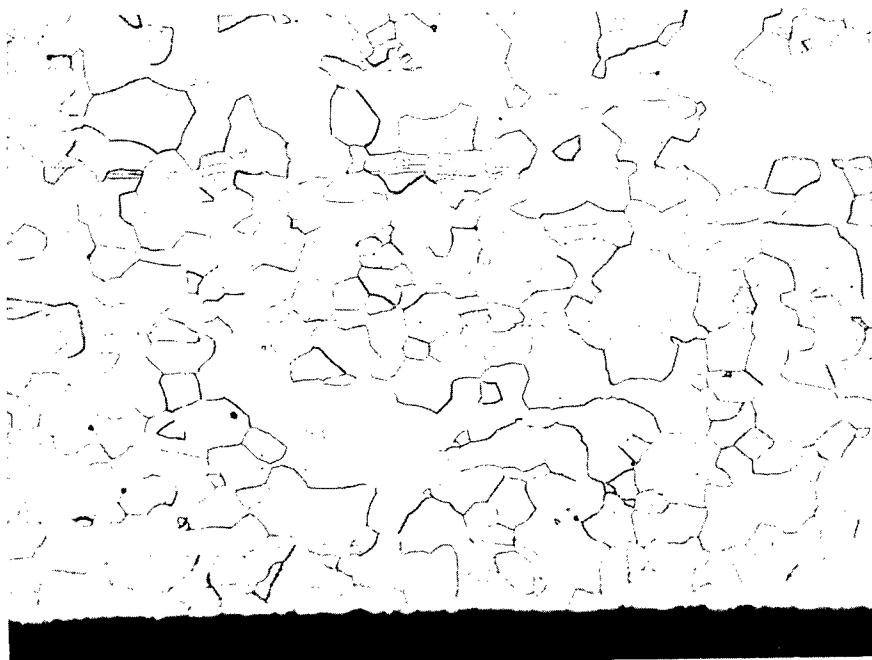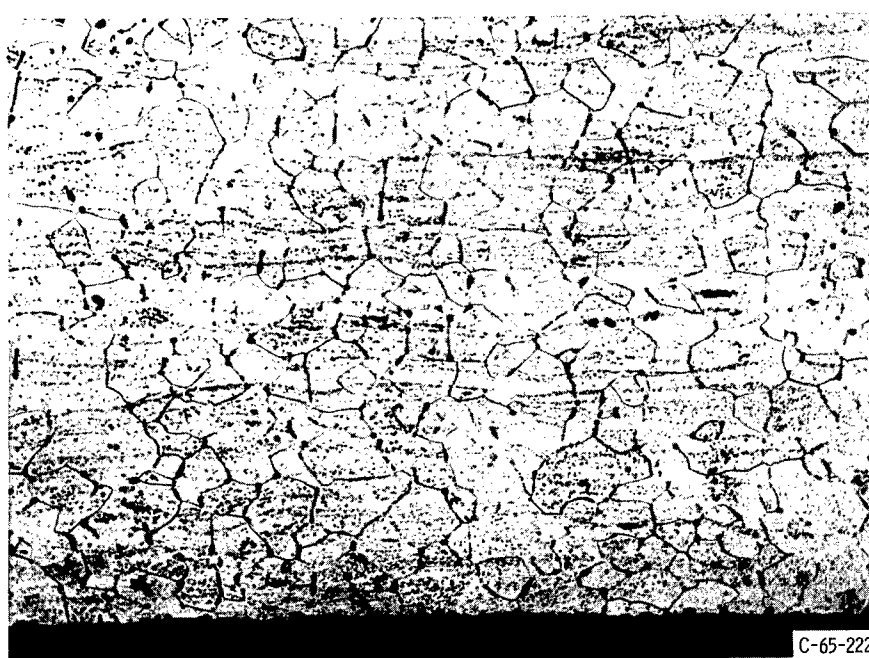
(b) After test.

Figure 16. - Comparison of microstructures of FS-85 before and after 2566-hour creep test at 2000° F and 8500 pounds per square inch in ultrahigh-vacuum unit. Etchant, equal parts of nitric and hydrofluoric acids modified with sulfuric acid, hydrogen peroxide, and acetic acid. X250.

Reference 9 states that creep curves similar in shape to those shown in figure 12 occur for unstable materials. In the present study of FS-85, portions of specimens that had been creep tested in the ultrahigh-vacuum creep units were metallographically examined after testing in an attempt to identify any observable metallurgical instability. For example, in figure 16, the microstructure of a specimen that had been creep tested for 2566 hours at 2000° F under a stress of 8500 pounds per square inch is compared with the initial recrystallized structure of the material before testing. A significant change in the microstructure was observed. The amount of second phase present in the microstructure of the creep-tested specimen greatly increased, while the grain size remained virtually unchanged at 0.020 millimeter. The formation of large, unidentified second-phase particles in this alloy, which was accompanied by significant softening (Knoop hardness decreased from 221 to 185), may be the type of metallurgical instability responsible for the unusual shape of the observed creep curves.

SUMMARY OF RESULTS

The high-temperature creep properties of a commercially available columbium alloy, FS-85, were characterized by short-time tests at pressures of 10^{-6} to 10^{-7} torr, and by long-time tests at pressures of 10^{-7} to 10^{-9} torr. The results of this study are as follows:

1. The alloy FS-85 exhibits moderate creep strength at 2000° F, but the creep rate increases rapidly with increasing temperature. For example, at a stress level of 10 000 pounds per square inch, FS-85 exhibited 1-percent creep in 775 hours at 2000° F; at 2200° F under the same stress, the alloy crept 1 percent in only 50 hours.

2. The Manson-Haferd linear parameter is moderately successful in predicting the long-time creep behavior of FS-85 at 2000° and 2200° F from relatively short-time tests over a wider range of temperatures. For example, the linear-parameter method predicts a time of 1360 hours to achieve a creep strain of 1 percent at 2200° F under a stress of 4000 pounds per square inch; the experimentally determined time was 1140 hours. Under other stress-temperature conditions, predicted and experimental times differ by a factor ranging from 1.05 to 1.7. When the scatter generally observed in high-temperature creep data is considered, the agreement of prediction and experiment is good, although not on the conservative side.

3. The linear parameter can be used to predict the allowable stress for FS-85 to limit creep strain at 2000° F to 1 percent in 10 000 hours, a value needed for preliminary design purposes. The predicted value is 5000 pounds per square inch.

4. Creep curves determined under conditions of ultrahigh-vacuum (10^{-8} to 10^{-9} torr), high temperature (2000° and 2200° F), and relatively low stress (4000 to 10 000

psi) generally exhibit a relatively unusual shape, with creep rate continuously increasing from the start of testing. The general absence of any lengthy period of linear creep behavior precludes the use of a steady-state creep rate as a design criterion for this alloy under such conditions.

Lewis Research Center,
　　　National Aeronautics and Space Administration,
　　　　　Cleveland, Ohio, April 14, 1965.

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